

CLAIMS

What is claimed is:

1. A method for making a disulfide, comprising the steps of:
 - (1) oxidation of a mercaptan with sulfur, wherein the oxidation reaction has a 1-10% molar excess of mercaptan to sulfur;
 - (2) removing hydrogen sulfide;
 - (3) reacting the product of step (2) remaining after the hydrogen sulfide removal with hydrogen peroxide; and
 - (4) removing water to less than 1 wt%.
2. The method of claim 1, wherein the mercaptan is β -mercaptoethanol and the disulfide is dithiodiglycol.
3. The method of claim 1, wherein the mercaptan is selected from the group consisting of C_2 to C_{20} alkyl mercaptans, cycloalkylmercaptans, functionalized mercaptans and acids and esters thereof.
4. The method of claim 1, wherein the oxidation reaction has a 3-5% molar excess of mercaptan to sulfur.
5. The method of claim 1, wherein the water is removed to less than 0.5 wt%.
6. The method of claim 1, wherein greater than 80% of the hydrogen sulfide is removed.
7. The method of claim 6, wherein greater than 90% of the hydrogen sulfide is removed.
8. The method of claim 1, wherein the removal of the hydrogen sulfide is by vacuum, nitrogen sparge or a combination thereof.

9. The method of claim 1, wherein the hydrogen peroxide in step (3) has a concentration of hydrogen peroxide in solution of between 5 wt% and 98 wt%.
10. The method of claim 1, wherein the hydrogen peroxide in step (3) has a concentration of hydrogen peroxide in solution of between 25 wt% and 70 wt%.
11. The method of claim 1, wherein the hydrogen peroxide in step (3) has a concentration of hydrogen peroxide in solution of between 27.5 wt% and 50 wt%.
12. The method of claim 1, wherein the water removal in step (4) is by vacuum stripping.
13. The method of claim 1, wherein the water removal in step (4) is by vacuum stripping with a nitrogen sparge.
14. The method of claim 1, wherein the water removal in step (4) is by a wiped film evaporator.
15. A product made by a process of claim 1.
16. A product made by a process comprising the steps of:
 - (1) oxidation of β -mercaptoethanol with sulfur, wherein the oxidation reaction has a 1-10% molar excess of β -mercaptoethanol to sulfur; and
 - (2) removing the hydrogen sulfide;wherein the product comprises greater than 87 wt% dithiodiglycol; less than 5 wt% trithiodiglycol; and less than 7 wt% unreacted β -mercaptoethanol.
17. The product of claim 16, wherein the product comprises greater than 92 wt% dithiodiglycol.

18. The product of claim 16, wherein the product comprises less than 3 wt% trithiodiglycol.
19. The product of claim 16, wherein the product comprises less than 5 wt% unreacted β -mercaptoethanol.
20. The method of claim 16, wherein the oxidation reaction has a 3-5% molar excess of mercaptan to sulfur.
21. The method of claim 16, wherein the removal of the hydrogen sulfide is by vacuum, nitrogen sparge or a combination thereof.
22. The method of claim 16, greater than 80% of the hydrogen sulfide is removed.
23. The method of claim 22, greater than 90% of the hydrogen sulfide is removed.
24. A product made by a process comprising the steps of:
 - (1) oxidation of β -mercaptoethanol with sulfur, wherein the oxidation reaction has a 1-10% molar excess of β -mercaptoethanol to sulfur;
 - (2) removing the hydrogen sulfide; and
 - (3) reacting the product of step (2) remaining after the hydrogen sulfide removal with hydrogen peroxide;wherein the residual β -mercaptoethanol in the product is less than 0.02 wt% mercaptan.
25. The method of claim 24, wherein the oxidation reaction has a 3-5% molar excess of mercaptan to sulfur.
26. The method of claim 24, wherein the removal of the hydrogen sulfide is by vacuum, nitrogen sparge or a combination thereof.

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27. The method of claim 24, wherein greater than 80% of the hydrogen sulfide is removed.
28. The method of claim 27, wherein greater than 90% of the hydrogen sulfide is removed.
29. The method of claim 24, wherein the residual water is less than 6 wt%.
30. The method of claim 24, wherein the residual water is less than 1 wt%.
25. The method of claim 24, wherein the residual water is less than 0.5 wt%.